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## PATENT ABSTRACTS OF JAPAN

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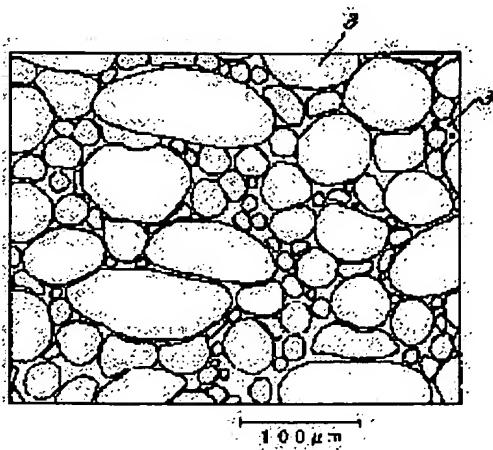
OTSUKA ISAMU

### (54) PRODUCTION OF AMORPHOUS SOFT MAGNETIC ALLOY POWDER MOLDED BODY

#### (57)Abstract:

PROBLEM TO BE SOLVED: To improve the thermal conductivity of raw material powder at the time of hot molding and to produce an amorphous soft magnetic alloy powder molded body in a short time.

SOLUTION: Raw material powder is previously subjected to preliminary molding and the obtnd. preliminarily molded body is housed in a hot molding die and is subjected to heating and pressurizing to mold an amorphous soft magnetic alloy powder compacted body. Concretely, the raw material powder composed of amorphous soft magnetic alloy powder 3, glass 30 in which the softening point is lower than the crystallization starting temp. of the amorphous soft magnetic alloy and a binding resin is pressurized in



JAPANESE [JP,11-256202,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF THE INVENTION  
TECHNICAL PROBLEM MEANS EXAMPLE  
DESCRIPTION OF DRAWINGS DRAWINGS

[Translation done.]

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CLAIMS

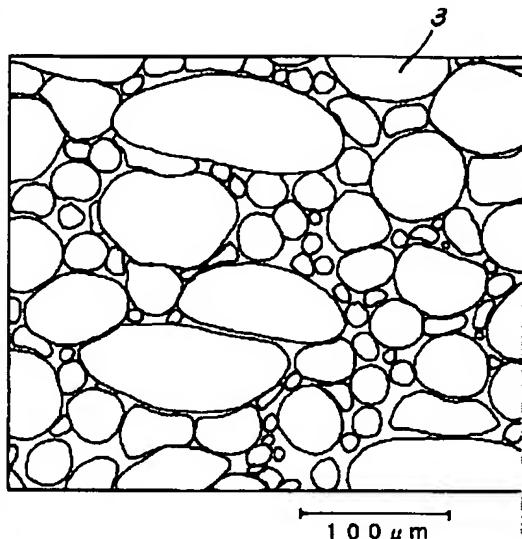
[Claim(s)]

[Claim 1] Amorphous soft magnetism alloy powder and glass with softening temperature lower than the crystallization initiation temperature of an amorphous soft magnetism alloy, Pressurize the raw material powder which consists of bending resin, and a preforming object is formed according to the binding capacity of bending resin. The manufacture approach of the amorphous soft magnetism alloy powder-molding object which is more expensive than the softening temperature of glass, carries out pressing of the acquired preforming object at temperature lower than the crystallization initiation temperature of an amorphous soft magnetism alloy, and is characterized by joining an amorphous soft magnetism alloy particle through glass.

[Claim 2] A preforming object is the manufacture approach of the amorphous soft magnetism alloy powder-molding object according to claim 1 characterized by being higher than the softening temperature of glass, holding into the metal mold beforehand heated by temperature lower than the crystallization initiation temperature of an amorphous soft magnetism alloy.

Drawing selection

Representative drawing



[Translation done.]

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## DETAILED DESCRIPTION

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### [Detailed Description of the Invention]

#### [0001]

[Field of the Invention] This invention relates to the manufacture approach of the powder-compacting Plastic solid of an amorphous soft magnetism alloy of having used low softening temperature glass as a binder.

#### [0002]

[Description of the Prior Art] It is known that the property which was excellent in respect of corrosion resistance, abrasion resistance, reinforcement, permeability, etc. is shown compared with a crystal ingredient, and the amorphous soft magnetism alloy is used as a magnetic material of the various devices in the electrical and electric equipment or electronic equipment. the relation top of the quenching process from which this amorphous soft magnetism alloy secures an amorphous condition, and its configuration -- general -- the shape of thin band-like one and a thin line -- or it is powdered. Therefore, in order to obtain the member of a predetermined configuration, after once grinding and making it powder also with the thing of the shape of thin band-like one or a thin line, it is necessary to pressurize and to fabricate in the condition of having heated to predetermined temperature.

[0003] By the way, the forming cycle of amorphous soft magnetism alloy powder must be performed at temperature lower than the crystallization initiation temperature of an alloy, in order to maintain the amorphous state of an alloy. However, alloy powder cannot be made to bulk-ize at this temperature. For this reason, the raw material powder which mixed the low glass powder of softening temperature to amorphous soft magnetism alloy powder is held in the molding die between heat, and the approach of forming an amorphous soft magnetism alloy powder-molding object is adopted by joining amorphous soft magnetism alloy particles for the glass which carried out hot forming and which was softened at temperature [ higher than the softening temperature of glass ] lower than the crystallization initiation temperature of an amorphous soft magnetism alloy as a binder.

#### [0004]

[Problem(s) to be Solved by the Invention] In the above-mentioned approach, since many openings exist between particles in the state of powder in case raw material powder is held in the molding die between heat and it heats to a predetermined molding temperature, overall thermal conductivity is small and the temperature gradient by the side of the wall surface of metal mold and a core tends to become large. In order to fabricate by heating raw material powder to homogeneity, the heating time for about about 20 - 40 minutes is needed, and productivity falls. Moreover, when manufacturing a thick different Plastic solid, according to a thick difference, temperature nonuniformity is produced to powder and a Plastic solid with a uniform property is not acquired. On the other hand, in order to aim at improvement in productivity, when metal mold tends to be heated more to an elevated temperature, a heat gain tends to be enlarged and it is going to shorten heating time, the temperature gradient by the side of a wall surface and a core has the problem by which amorphous nature will be spoiled for the temperature of the powder by the side of a wall surface exceeding the crystallization initiation temperature of an amorphous soft magnetism alloy, when it becomes still larger and a core side reaches molding temperature.

[0005] The purpose of this invention is enabling it to manufacture an amorphous soft magnetism alloy powder-molding object for a short time while aiming at improvement in the thermal conductivity of the raw material powder at the time of hot forming.

[0006]

[Means for Solving the Problem] In order to solve the above-mentioned technical problem, this invention forms an amorphous soft magnetism alloy powder-molding object by fabricating raw material powder beforehand, holding the acquired preforming object in the molding die between heat, and heating and pressurizing it. Specifically Amorphous soft magnetism alloy powder and glass with softening temperature lower than the crystallization initiation temperature of an amorphous soft magnetism alloy, Pressurize the raw material powder which consists of bending resin in a reserve molding die, form a preforming object according to the binding capacity of bending resin, and the acquired preforming object in the molding die between heat An amorphous soft magnetism alloy powder-molding object is manufactured by being higher than the softening temperature of glass, carrying out pressing at temperature lower than the crystallization initiation temperature of an amorphous soft magnetism alloy, and joining an amorphous soft magnetism alloy particle through glass.

[0007] The molding die between heat in which a preforming object is held is higher than the softening temperature of glass beforehand, and it is desirable to heat to temperature lower than the crystallization initiation temperature of an amorphous soft magnetism alloy.

[0008]

[Function and Effect] Raw material powder is put into the metal mold for preforming, by pressurizing, raw material powder is bound with bending resin, and a preforming object is formed. An amorphous soft magnetism alloy particle is joined through glass by holding the acquired preforming object in the molding die between heat, and giving heating and pressurization. Since it is precise compared with the thing of a powder condition, a preforming object has large thermal conductivity. Therefore, even if it makes a programming rate quick, the homogeneity of overall temperature can be secured, and it can prevent that it will be in overheating partially. For this reason, the time amount which hot forming takes can also be shortened. Bending resin transpires with heating at the time of hot forming.

[0009] If the metal mold for hot forming is beforehand heated to predetermined molding-temperature extent, the time amount which hot forming takes can be shortened further.

[0010]

[Embodiment of the Invention] The amorphous soft magnetism alloy, glass, and bending resin which are used for this invention are explained.

As an amorphous soft magnetism alloy amorphous soft magnetism alloy, alloys, such as Fe systems (Fe-Si-B etc.) and Co systems (Co-Fe-Si-B etc.), can be mentioned. The crystallization initiation temperature of these alloys is usually around about 500 degrees C. The powder of an amorphous soft magnetism alloy can be obtained by well-known various approaches. For example, the high-speed rotation stream atomizing method and the rotation liquid atomizing method can be illustrated. As for the particle size of amorphous soft magnetism alloy powder, it is desirable to be referred to as about 250 micrometers or less, and about about 100-150 micrometers is suitable for mean particle diameter.

[0011] The thing of temperature with softening temperature lower about about 80-400 degrees C than the crystallization initiation temperature of said amorphous soft magnetism alloy is used for glass glass. For example, it is desirable to use that whose softening temperature is about 100-400 degrees C. This is for giving width of face to the molding temperature of hot forming. As the seed glass ingredient, low softening temperature glass, such as borate system glass (PbO and B<sub>2</sub>O<sub>3</sub>) of lead oxide content and ternary system glass which made ZnO or SiO<sub>2</sub> mix in this, can be mentioned. As for the amount of mixing of glass, considering as 3 - 20vol% is desirable, and it should just set it up according to desired permeability within the limits of this. If there are too few amounts of mixing of glass, while it will become difficult for a binder operation to be insufficient and to make amorphous soft magnetism alloy powder bulk-ize, there is a possibility that the insulation between amorphous soft magnetism alloy particles may fall. On the other hand, although a mechanical strength will become large if there are too

Q, 20  
and  
example

many amounts of mixing of glass, the amount which the amorphous soft magnetism alloy in a Plastic solid occupies becomes small, and there is a possibility that it may become impossible to secure sufficient magnetic properties.

[0012] Even after binding bending resin at the time of preforming where eburnation of the raw material powder is carried out to some extent, and taking out from preforming metal mold, unless the excessive force is applied, the resin ingredient which has the binding property of extent which can maintain a predetermined configuration is used. As the seed bending resin ingredient, organic substance system binders, such as an epoxy resin, PVA and a wax or elastic phenol resin, and acrylic resin, can be mentioned.

[0013] Raw material powder is produced from the above-mentioned amorphous soft magnetism alloy, glass, and bending resin. As raw material powder, three kinds of things as follows can be illustrated.

\*\* Powder which mixed bending resin with amorphous soft magnetism alloy powder and glass powder.

\*\* The composite particle which performed glass coating to the front face of amorphous soft magnetism alloy powder, and powder which mixed bending resin.

\*\* Powder which performed coating of glass and bending resin to the front face of amorphous soft magnetism alloy powder.

[0014] Hereafter, the manufacture approach of each raw material powder of \*\* - \*\* is explained in order.

\*\* The raw material powder of raw material powder \*\* consists of amorphous soft magnetism alloy powder, glass powder, and bending resin. A powder-like thing may be used for bending resin and the thing of liquefied or gel may be used for it. Drawing 1 is drawing showing typically the raw material powder which consists of amorphous soft magnetism alloy powder (3), glass powder (32), and bending powder-like resin (34). When using bending powder-like resin, amorphous soft magnetism alloy powder, glass powder, and bending resin powder are prepared, and raw material powder is prepared by mixing these. In addition, when the particle diameter of amorphous soft magnetism alloy powder is about 100-150 micrometers, the particle diameter of glass powder is set to about 3-7 micrometers, and, as for the particle diameter of bending resin powder, it is desirable to be referred to as about 0.1-10 micrometers. Moreover, when the particle diameter of amorphous soft magnetism alloy powder is about 50-100 micrometers, the particle diameter of glass powder is set to about 1-5 micrometers, and, as for the particle diameter of bending resin powder, it is desirable to be referred to as about 0.1-5 micrometers. When using the bending resin of liquefied or gel, paste-like raw material powder is produced by adding the bending resin of liquefied or gel, mixing amorphous soft magnetism alloy powder and glass powder, or mixing these powder. In addition, it is desirable to perform mixing with powder and bending resin under an inert gas ambient atmosphere or a vacuum.

[0015] \*\* Beforehand, the raw material powder of raw material powder \*\* mixes bending resin (34) with the composite particle powder which performed glass coating (36) on the front face of amorphous soft magnetism alloy powder (3), and is prepared on it. Drawing 2 is drawing showing this raw material powder typically. Composite particle powder is producible using the powder coating equipment shown in drawing 4. Drawing 4 is drawing showing the powder coating equipment used for producing a composite particle, and is the sectional view cut towards intersecting perpendicularly with an axial center in the location near the single-sided edge of the cylindrical cup (10) of equipment. When drawing 4 is referred to, the interior of the cylindrical cup (10) which can be sealed The 1st arm (12) has projected from the boss (11) fixed to the revolving shaft (20) to radial. At the tip of this 1st arm (12) The press member (14) of the boiled-fish-paste form extended to the shaft orientations of a container (10) is formed, and in order to enable it, as for this press member (14), to press and compress powder, spacing predetermined in the apical surface and container inside is opened. From a boss (11), the scraper (18) with which the 2nd arm (16) has projected to radial in the opposite direction, and turns into said 1st arm (12) from the plate of a long and slender configuration towards the shaft orientations of a container (10) at the tip of the 2nd arm (16) is formed, and this scraper is arranged so that powder (22) can be scratched, and a container inside may be contacted mostly. In addition, a container (10) can be made into the bottom of a vacuum or an inert gas ambient atmosphere environment. The revolving shaft (20) is

coordinated with the rotation driving gear (not shown), and carries out high-speed rotation of the 1st arm (12) and the 2nd arm (16) with a revolving shaft (20). In addition, as for drawing in case a scraper (18) is located in the bottom, and (b), the press member (14) of (a) is drawing when being located in the bottom among drawing 3.

[0016] Composite particle powder is prepared in the following way using this powder coating equipment. The amorphous soft magnetism alloy powder (22) and glass powder (22) which were fed into the container (10) are scratched with a scraper (18), are taken, and are agitated, next are pushed against the inner skin of a container (10) by the press member (14), and receive a powerful compression friction operation. As a result of surface fusion taking place between an amorphous soft magnetism alloy particle and a glass particle and glass particles' welding by repeating these operations at high speed, the composite particle by which the front face of an amorphous soft magnetism alloy particle (3) was covered with the layer (36) of glass is obtained (refer to drawing 2 ). As for the thickness of a glass layer, it is desirable to make it about 3 micrometers of maxes. It is because there is a possibility of it being easy to produce the chip omission of a glass layer if 3 micrometers is exceeded, and thickness becoming an ununiformity, and spoiling insulation.

[0017] In addition, although production of this composite particle is performed under an inert gas ambient atmosphere or a vacuum for antioxidantizing, since the gas molecule which checks junction between solid-state-solid-states does not exist, and compound-ization of a particle is promoted, it is [ be / it / if / it carries out under a vacuum, ] more desirable.

[0018] It is the same point as \*\*, and it is mixed with powder and the bending resin of liquefied or gel, and raw material powder is prepared the end of composite powder it was obtained.

[0019] \*\* The raw material powder of raw material powder \*\* consists of a composite particle by which coating of glass and bending resin was performed to the front face of amorphous soft magnetism alloy powder. Drawing 3 is drawing showing this raw material powder typically. The glass to an amorphous soft magnetism alloy particle, and coating of bending resin \*\* If it can carry out with the powder coating equipment used on the occasion of production of raw material powder, amorphous soft magnetism alloy powder (22), glass powder (22), and bending resin powder (22) are fed into a container (10) and equipment is operated. The composite particle by which surface fusion took place between an amorphous soft magnetism alloy particle, a glass particle, and bending resin powder, and the front face of an amorphous soft magnetism alloy particle (3) was covered with the compression friction operation in the layer (38) of glass and bending resin is obtained. As for the thickness of the coating layer formed in the front face of an amorphous soft magnetism alloy particle, it is desirable to make it about 3 micrometers of maxes. It is because there is a possibility of it being easy to produce the chip omission of a coating layer if 3 micrometers is exceeded, and thickness becoming an ununiformity, and spoiling insulation.

[0020] The preforming object with which powder was combined with bending resin is formed by filling up with and carrying out pressing of the raw material powder prepared by the point of the formation above of a preforming object to a reserve molding die. Although it is desirable to perform this shaping in ordinary temperature, according to the degree of softening of resin, it can heat moderately and can also carry out (however, heating at the time of pressurization is made into temperature lower than the softening temperature of glass even in such a case). By performing preforming, the bulk Plastic solid which carried out eburnation to some extent is acquired. Even if it picks out this preforming object from preforming metal mold, that configuration is held unless the excessive force is applied.

[0021] What is necessary is just to carry out the temperature up of the preforming object to the temperature to which bending resin transpires, when the bending resin contained in a preforming object needs to be removed before production of a Plastic solid. As for a temperature up, it is desirable to carry out under an inert gas ambient atmosphere or in a vacuum.

[0022] the formation profit \*\*\*\* preforming object of a Plastic solid -- hot forming -- public funds -- it holds in a mold and a Plastic solid is produced by giving heating and pressurization. as the pressing approach -- hot pressing and the isostatic pressing (HIP) between heat -- law etc. is employable. The temperature at the time of shaping is higher than the softening temperature of glass, and is adjusted to temperature lower than the crystallization initiation temperature of an amorphous soft magnetism alloy.

For example, when Fe system amorphous soft magnetism alloy of Fe-Si-B with a crystallization initiation temperature of about 500 degrees C and borate system glass of about 320-400 degrees C of softening temperatures are used, the pressing can be performed the condition for [ temperature / of about 400-480 degrees C /, about one to 2 pressure GPa, and pressurization time amount ] about 1 minute. In addition, even if it does not transpire beforehand the bending resin contained in a preforming object, if a preforming object is heated, since the bending resin in a reserve Plastic solid transpires, to the acquired Plastic solid, bending resin will hardly remain.

[0023] If a preforming object is heated to temperature higher than the softening temperature of glass, it will soften and glass will present a fluidity. The glass which presented the fluidity invades without a clearance between amorphous soft magnetism alloy particles by continuing pressurization in this condition. By functioning also as an insulating material between amorphous soft magnetism alloy particles, there are few power losses by the overcurrent and the acquired Plastic solid has the advantage that decline in the permeability in a RF field is small while glass functions as a binder and it gives a desired mechanical strength to a Plastic solid.

[0024] When using the amorphous soft magnetism alloy powder-molding object of this invention for the core of a choke coil, a flyback transformer, etc., it is desirable to machine further, to heat again in the last configuration in finishing and a temperature requirement [ lower than the crystallization initiation temperature of an amorphous soft magnetism alloy and ] higher than the softening temperature of glass, and to perform distorted picking heat treatment. About 10 - 20 minutes are suitable for the heating holding time. Even if a mechanical distortion arises to amorphous soft magnetism alloy powder by performing such distorted picking heat treatment at the time of pressing, distortion is removed where the restraint from glass is removed by being heated after that by the temperature to which glass exceeds the softening temperature again. Consequently, since the property which an amorphous soft magnetism alloy originally has since the magnetic properties therefore spoiled distorted are recovered is maintained as much as possible also in a Plastic solid, that core can possess outstanding magnetic properties.

[0025]

[Example] The concrete example of an amorphous soft magnetism alloy powder-molding object is explained.

As <preparation of raw material powder> raw material powder \*\* amorphous soft magnetism alloy powder, Fe78Si nine B13 (about 100 meshes of maximum grain sizes), They are PbO, B-2O<sub>3</sub>, and SiO<sub>2</sub> system glass (the mean particle diameter of about 10 micrometers) as glass powder. Prepare a powder-like epoxy resin (about 100 meshes of maximum grain sizes) as 360 degrees C of softening temperatures, and bending resin, and weighing capacity is carried out so that it may become epoxy resin powder 10vol% glass powder 10vol% amorphous soft magnetism alloy powder 80vol%. It supplied to the ball mill and raw material powder \*\* was prepared by mixing for 24 hours.

[0026] As raw material powder \*\* amorphous soft magnetism alloy powder, Fe78Si nine B13 (about 100 meshes of maximum grain sizes), Prepare PbO, B-2O<sub>3</sub>, and SiO<sub>2</sub> system glass (mean particle diameter of about 10 micrometers, 360 degrees C of softening temperatures) as glass powder, and weighing capacity is carried out so that it may become glass powder 10vol% amorphous soft magnetism alloy powder 90vol%. It supplied to the powder coating equipment shown in drawing 4, and the powder with which the layer of glass becomes the front face of the particle which makes an amorphous soft magnetism alloy a mother particle from the composite particle by which covering formation was carried out was produced. As for the obtained composite particle, the mean particle diameter of an amorphous soft magnetism alloy particle of the thickness of about 75 micrometers and a glass layer was about 2 micrometers. Using a powder-like epoxy resin (about 100 meshes of maximum grain sizes) as bending resin, said composite particle powder 90vol% and bending resin powder 10vol% were supplied to the ball mill, and raw material powder \*\* was prepared by mixing for 24 hours.

[0027] As raw material powder \*\* amorphous soft magnetism alloy powder, Fe78Si nine B13 (about 100 meshes of maximum grain sizes), They are PbO, B-2O<sub>3</sub>, and SiO<sub>2</sub> system glass (the mean particle diameter of about 10 micrometers) as glass powder. Prepare a powder-like epoxy resin (about 100 meshes of maximum grain sizes) as 360 degrees C of softening temperatures, and bending resin, and

weighing capacity is carried out so that it may become epoxy resin powder 10vol% glass powder 10vol% amorphous soft magnetism alloy powder 80vol%. It supplied to the powder coating equipment shown in drawing 4, and raw material powder \*\* to which the layer of glass and an epoxy resin becomes the front face of the particle which makes an amorphous soft magnetism alloy a mother particle from the composite particle by which covering formation was carried out was prepared. As for the obtained composite particle, the mean particle diameter of an amorphous soft magnetism alloy particle of the thickness of the layer of about 85 micrometers, glass, and an epoxy resin was about 3 micrometers.

[0028] The preforming metal mold for <formation of preforming object> cold pressing (product made from SKD11) was filled up with raw material powder, it pressurized by 500MPa under the ordinary temperature ambient atmosphere, and the preforming object with a diameter [ of 20mm ] and a height of 8mm was produced. When the acquired preforming object was picked out from preforming metal mold and observed, composite particles joined together with bending resin, and they had become the bulk Plastic solid which carried out eburnation to some extent. Even if it picked out this preforming object from preforming metal mold, the mold collapse of it was not carried out, but it was maintaining the predetermined configuration.

[0029] produced preforming -- in order to transpire the bending resin contained inside of the body, among the vacuum, on condition that the programming rate of 45 degrees C / min, the preforming object was held for 10 minutes and carried out the temperature up to about 450 degrees C. consequently, preforming -- since the glass formed in the composite particle front face starts softening and joins particles instead of bending resin while bending resin in the living body transpires, the configuration of a preforming object is held and the reinforcement is also maintained.

[0030] <formation of a Plastic solid> -- said preforming object was held in the hot-forming metal mold which heats the hot-forming metal mold for hot pressing (product made from SKD61) at about 450 degrees C, and was heated beforehand, it was pressurized for 0.5 minutes by 1000MPa under the 450-degree C vacuum ambient atmosphere, and the Plastic solid with a diameter [ of 20mm ] and a height of 6mm was produced. When the acquired Plastic solid was picked out from hot-forming metal mold and observed, as shown in drawing 5, it was joined and the Plastic solid was bulk-sized while amorphous soft magnetism alloy powder (3) was insulated through glass (30). When the relative density of each Plastic solid formed from raw material powder \*\*, \*\*, and \*\* was measured, it was 98%, 99%, and 98%, and was a precise Plastic solid altogether, respectively. In addition, "relative density" searches for a Plastic solid as a ratio of the actual weight to the weight when assuming completely that it is a precise object, and the weight of a completeness precise object is the value calculated based on the mixing ratio of amorphous soft magnetism alloy powder and glass powder.

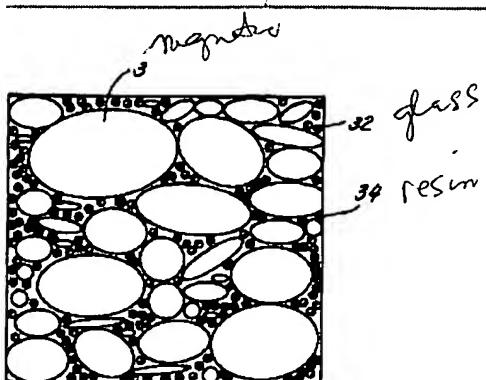
[0031] <Finish> Finish-machining according the acquired Plastic solid to machining was performed, and finishing and magnetic properties were measured in the ring-like core configuration. Consequently, the eddy current generated between particles was controlled, it had permeability suitable as a core for RFs, and frequency characteristics were also good cores.

[0032] Explanation of the above-mentioned example is for explaining this invention, and it should not be understood so that invention of a publication may be limited to a claim or the range may be \*\*\*\*(ed). Moreover, deformation various by technical within the limits given not only in the above-mentioned example but a claim is possible for each part configuration of this invention.

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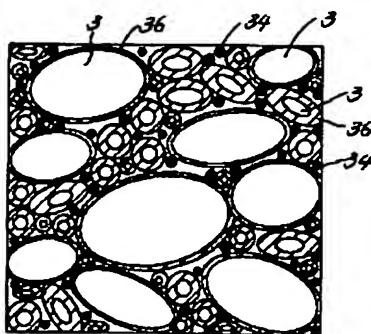
Drawing selection drawing 1



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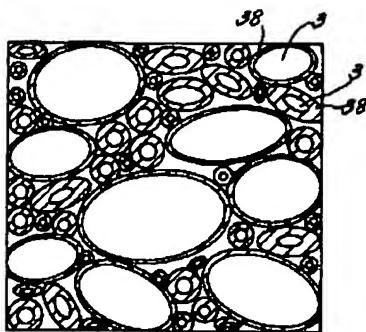
[Translation done.]

Drawing selection drawing 2



[Translation done.]

Drawing selection drawing 3



[Translation done.]